

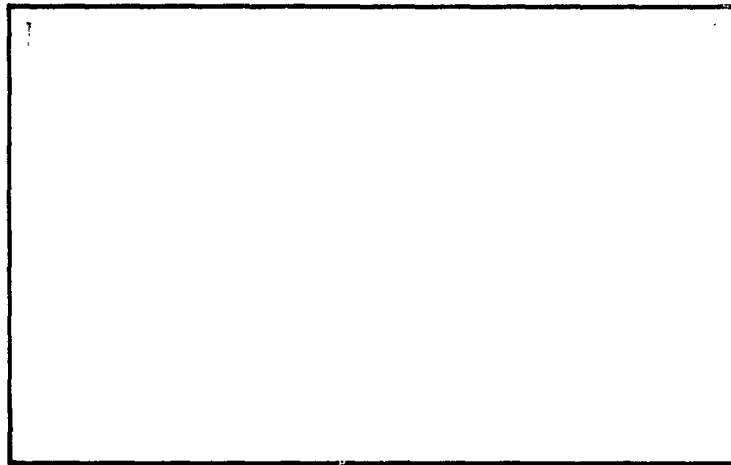
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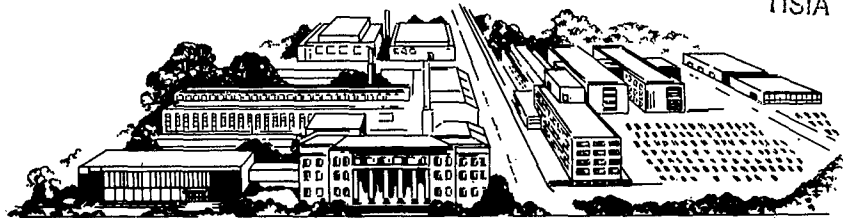
## RESEARCH REPORT

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THIRD QUARTERLY REPORT

on

MECHANISM OF WATER ABSORPTION IN  
GLASS-REINFORCED PLASTICS

to

DEPARTMENT OF THE NAVY  
BUREAU OF SHIPS

April 1, 1963

by

D. W. McNeil, Bailey Bennett, and R. I. Leininger

Contract No. NObs 86871  
Project No. SR-007-03-04  
Task No. 1008  
BuShips No. 98-1008-10

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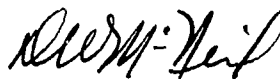
Gentlemen:

Enclosed is our third quarterly report on your project, "Mechanism of Water Absorption in Glass-Reinforced Plastics". This report covers activities from January 1 to April 1, 1963.

Experimental work has now progressed to the stages where a tentative explanation of the water-absorption phenomena may be proposed. This picture will be complete once the mechanical testing and diffusion experiments have been completed.

Any questions or comments pertinent to this work will be welcomed.

Very truly yours,



D. W. McNeil  
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# MECHANISM OF WATER ABSORPTION IN GLASS-REINFORCED PLASTICS

by

D. W. McNeil, Bailey Bennett, and R. I. Leininger

## INTRODUCTION

It has been observed that prolonged exposure of glass-reinforced plastics to water is usually accompanied by a resulting deterioration in their mechanical properties. This program is designed to obtain a better understanding of the mechanisms associated with the problem. Once such knowledge is at hand, it can point the way for appropriate correctional steps to be taken.

This report covers work completed between January 1 and April 1, 1963.

## SUMMARY

During the third quarter, work was evenly distributed between the two phases of this program. In the first of these, activity was primarily directed toward obtaining diffusion measurements of several hydrophilic polymers. These measurements, on model systems, will later be compared with similar values obtained at high pressures in order to establish the role of hydrostatic variables in the diffusion of a liquid into a solid polymer. The weight-gain experiments already indicate that diffusion phenomena in water-epoxy resin systems are essentially unaffected by hydrostatic pressures up to 10,000 psi.

Studies in the second phase have included electron-microscope and mechanical evaluations of glass-reinforced epoxy cylinders. Research with the electron microscope has indicated that high pressures may be the cause of flaws and other imperfections which allow the immersion medium to come into contact with the fibers below. As a result, the moisture may penetrate along the resin-glass bond and cause a decrease in flexural strength. Mechanical tests are being utilized to confirm this latter effect.

Recent mechanical tests on neat resin specimens show them to be much less sensitive to water absorption than the same resin reinforced with glass. This behavior points up the need for more intensive study of composites, particularly of interfacial effects.

## EXPERIMENTAL WORK

### Diffusion Measurements

It has been shown that water definitely diffuses into epoxy resin, but very slowly. A still more pronounced effect has been observed with glass-reinforced epoxy resin immersed at high hydrostatic pressures. In these latter studies, the indications are that pressure has little if any effect on the rate of water absorption.

One way to check the general effect of pressure as a factor in water absorption of resin is to use model systems to speed up the experimental procedure. Should pressure be shown to be a factor in the rate of water absorption, it will then be confirmed with the more water-resistant epoxy systems.

Four hydrophilic polymers were selected as model systems for experiments to determine the effects of hydrostatic pressure on diffusion rates:

- (1) Partially crosslinked gelatin (water insoluble)
- (2) Cellophane (a water-insoluble membrane used for dialysis)
- (3) Polyvinyl pyrrolidone
- (4) Gelatin (water soluble).

The diffusion of distilled water into each of these polymers was measured by using the diffusimeter and techniques described in the second quarterly report.\* Since all of these systems are structurally quite weak, it was necessary to design and fabricate a special clamping jig to hold the interferometer plates in alignment during the experiment. This device consists of an outer casing having six jackscrews at one end and a threaded nut at the other. The sample is placed between the silvered surfaces of the plates and inserted into the jig. After the nut is in place, the jackscrews are adjusted to give approximate parallelism (as observed from the interference-fringe system) of the plates. A thrust ring between the jackscrews and interferometer prevents mechanical damage to the flats during subsequent adjustment.

Initial experiments were conducted at atmospheric pressure, using a distilled-water immersion medium. Both distance-time and concentration-distance curves were made from interferograms taken at random intervals during each experiment. Each polymer was run at least three times to minimize the possibility of misinterpretation of data during subsequent pressure runs.

A second series of experiments was attempted using sea water (sp gr 1.023). However, no data were obtained because of the corrosive action of this material on the aluminum reflecting surfaces of the optical flats. The fringe patterns within the water film rapidly disappeared, thereby destroying the "reference" pattern necessary for subsequent calculations.

\* Second Quarterly Report, "Mechanism of Water-Absorption in Glass-Reinforced Plastics", from Battelle to Department of the Navy, Bureau of Ships (January 1, 1963).

It was originally anticipated that the evaporated-aluminum reflecting surfaces of the interferometer plates could be protected by depositing a thin layer of silicon monoxide on the surface. This procedure gives excellent results with distilled water, but not with sea water. Similar data for a sea-water system may, however, be obtained by using a reflecting film of rhodium (an inert metal less reactive than hydrogen).

Similar experiments with the same polymer systems are now being conducted at elevated pressures. These data will be included in the fourth quarterly report.

#### Electron Microscopy

Several samples were prepared for the specific purpose of examination with the electron microscope before and after subjecting them to hydrostatic pressure. These samples consisted of six resin cylinders and two filament-wound cylinders. They were selected from a large number of specimens on the basis of surface cleanliness and freedom from flaws.

A 1 x 10-mm strip along the length of each cylinder was outlined with indelible ink, and cellulose acetate surface replicas were prepared from this area. The samples were then placed in individual containers to prevent mechanical damage and subjected to a hydrostatic pressure of 10,000 psi for 63 days. At the end of this period, surface replicas were again prepared from the same areas. These replicas were examined in a JEM-5Y electron microscope and photographs obtained of significant areas. Subsequent enlargement of these photos gave magnifications ranging from 8,750X to 17,500X. Four of the most significant of the micrographs prepared from surface replicas along with the interpretation of each are presented in the following section of the report.

#### Interpretation

Figure 1 shows the surface texture of a resin cylinder prior to exposure to water at high pressure. Aside from a typical slight roughness and occasional scratches, no other structures were visible in any of the replicas prepared from this sample. Figure 2 shows a typical crack observed in a later replica from approximately the same area. These cracks were observed only after the cylinders had been exposed to high-pressure water. They were randomly scattered over the surface of the test cylinders.

Of additional interest, but of unknown significance, is the apparent change in surface texture during exposure. In almost all fields examined, the surface of the test cylinders was noticeably smoother after exposure. Initially, one might assign this to a slight swelling of the polymer which could accompany the diffusion of moisture into that polymer.

Figure 3 is the initial replica of the exterior surface of a capped filament-wound cylinder.\* This particular cylinder was covered with an outer resin layer approximately 1/32 inch thick and was selected on the basis of its freedom from gross surface irregularities. Although occasional small cracks were observed in the initial replica, they were relatively few in number, the major portion of the area examined having the typical appearance of Figure 3. After immersion at 10,000 psi, cracks such as those shown in

\*See First Quarterly Report, "Mechanism of Water Absorption in Glass-Reinforced Plastics", from Battelle to Department of the Navy, Bureau of Ships (October 1, 1962).



17,500X

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FIGURE 1. ELECTRON MICROGRAPH OF UNEXPOSED SURFACE  
OF RESIN CYLINDER

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17,500X

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FIGURE 2. ELECTRON MICROGRAPH OF APPROXIMATE AREA IN FIGURE 1  
AFTER EXPOSURE TO HIGH-PRESSURE WATER

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FIGURE 3. ELECTRON MICROGRAPH OF UNEXPOSED SURFACE OF FILAMENT-WOUND CYLINDER

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Figure 4 were fairly common. Of a total of eight fields photographed at random, six present the characteristic appearance of cracks. No attempt was made similarly to analyze the inside surfaces of these cylinders, as there were innumerable small flaws which could lead to difficulties in interpretation.

### Immersion Studies

Weight-gain measurements were continued from the preceding quarter on a variety of sample types. These consisted of both cast resin and filament-wound cylinders.

#### Cast Resin Cylinders

After almost 4,000 hours' immersion at both atmospheric and elevated pressures, no leakage has been observed in any of the cast resin cylinders, although crazing originating at the inner surface of those samples under pressure has become quite pronounced. Both the control samples immersed at atmospheric pressure and the samples exposed to hydrostatic pressure present almost identical weight-gain curves (see Figure 5). These data indicate that elevated hydrostatic pressures up to 10,000 psi do not appreciably affect the rate of diffusion of moisture in a typical cured epoxy-resin system.

#### Filament-Wound Cylinders

Also in Figure 5 is the moisture-absorption curve obtained for a group of capped filament-wound cylinders. The seemingly lower rate of absorption is rather deceptive. Since the filament-wound cylinders are approximately 80 per cent glass, the amount of water absorbed would be five times greater when calculated on the basis of weight per gram of resin. This is assuming, however, that all of the resin has become saturated. The slope of the curves indicates that this is not the case. These data will therefore be recomputed when diffusion measurements have been completed on epoxy-resin films.

Figure 6 shows the absorption curves obtained from open sections of filament-wound cylinders. Both the controls and those exposed to pressure are of similar size and composition. No explanation is attempted for the apparently greater absorption exhibited by the controls.

In all immersion studies, a soluble fluorescent dye was incorporated in the immersion medium. Upon failure\* of any of the capped filament-wound cylinders, the sample was removed from test. The sample was then washed, mounted, and polished prior to subsequent examination with ultraviolet light and by microscopic means. In several instances, the fluorescence of the dye revealed that water had penetrated along interlaminar flaws as well as entered the cylinder cavity by causing debonding of the adhesive (epoxy resin) at the caps.

\* By failure is meant leakage at the caps.





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FIGURE 4. ELECTRON MICROGRAPH OF EXPOSED SURFACE  
OF FILAMENT-WOUND CYLINDER

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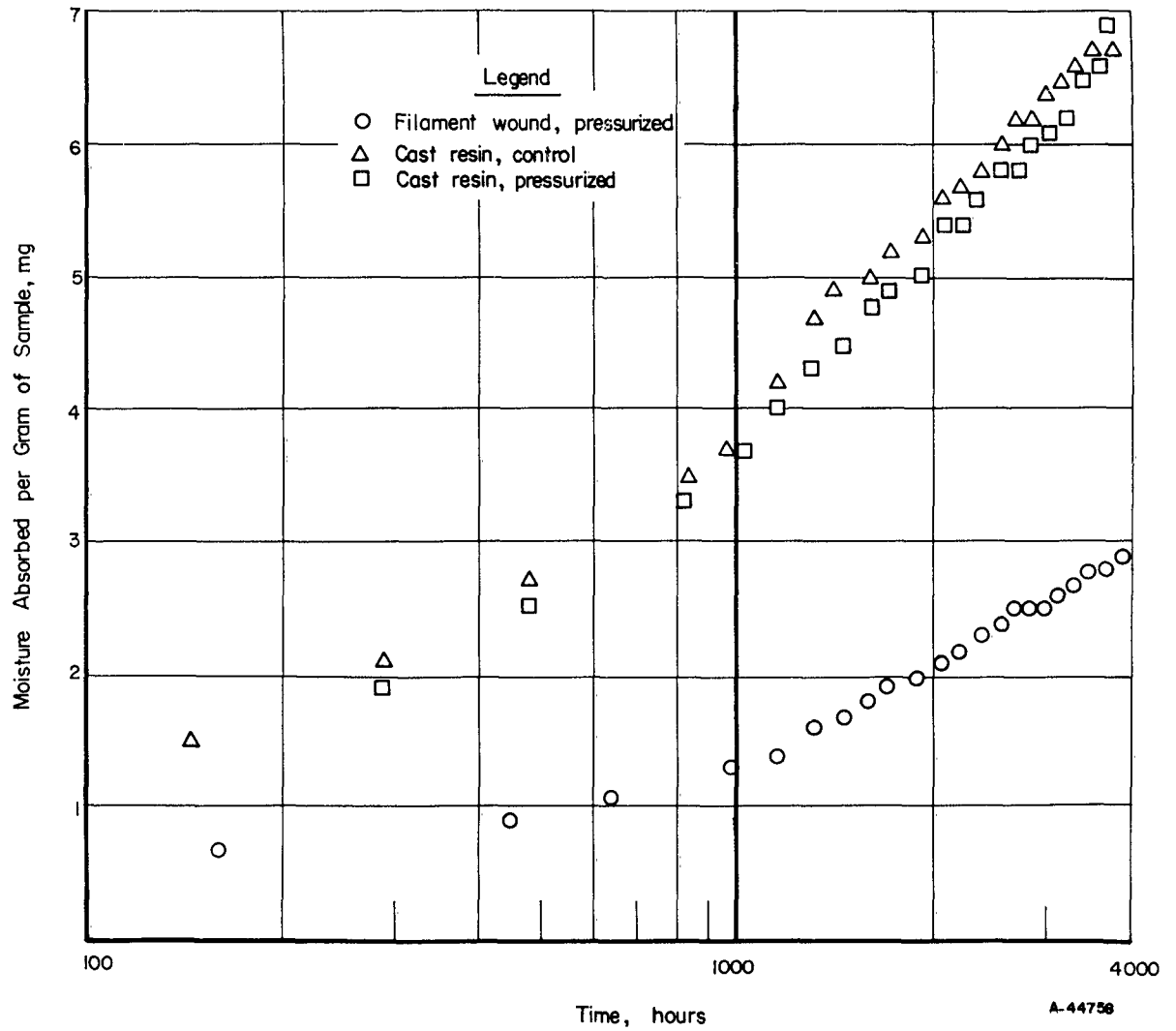


FIGURE 5. MOISTURE ABSORPTION OF 1-INCH, CAPPED, FILAMENT-WOUND AND CAST RESIN CYLINDERS

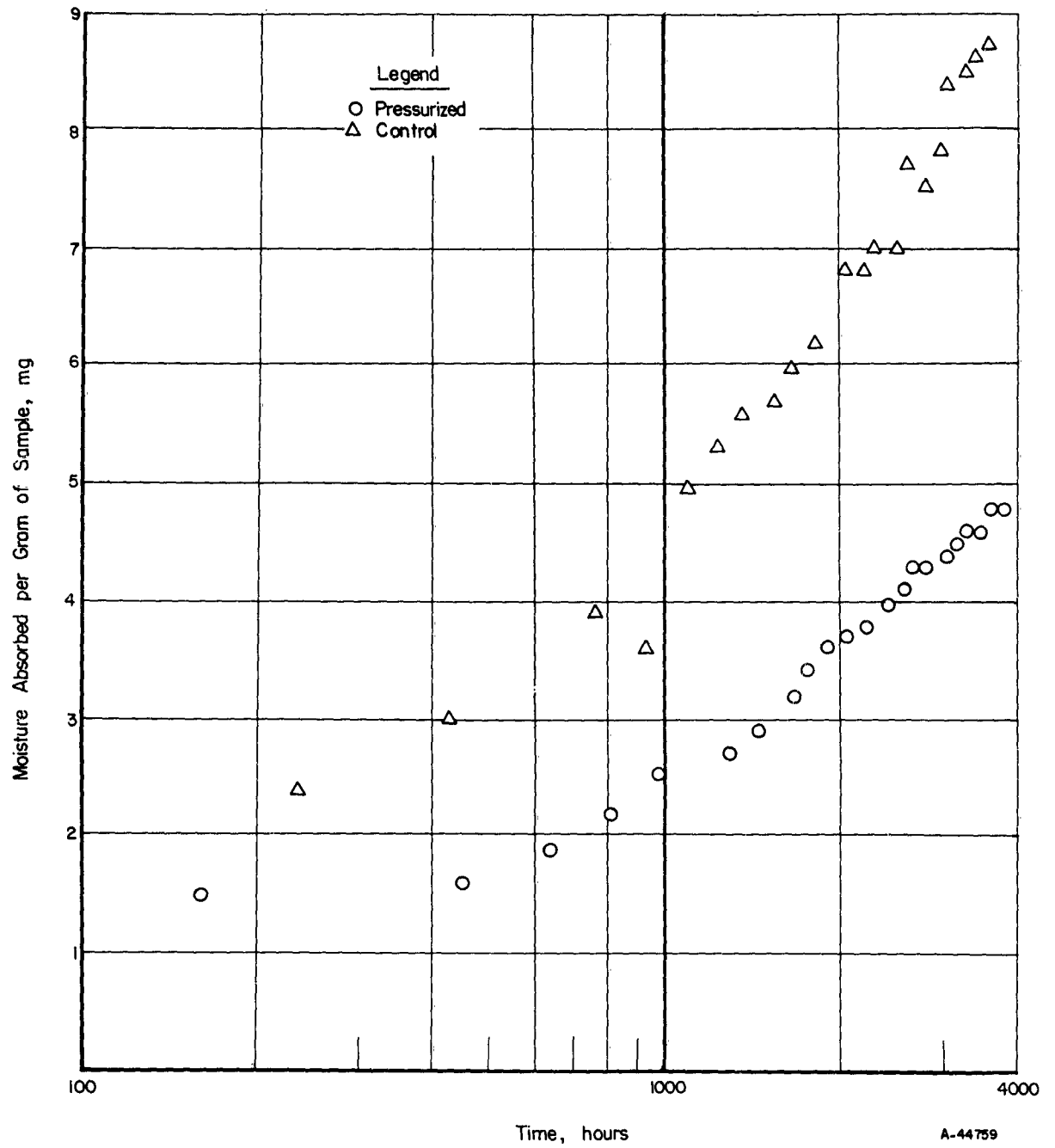


FIGURE 6. MOISTURE ABSORPTION OF 1-INCH, FILAMENT-WOUND OPEN CYLINDERS

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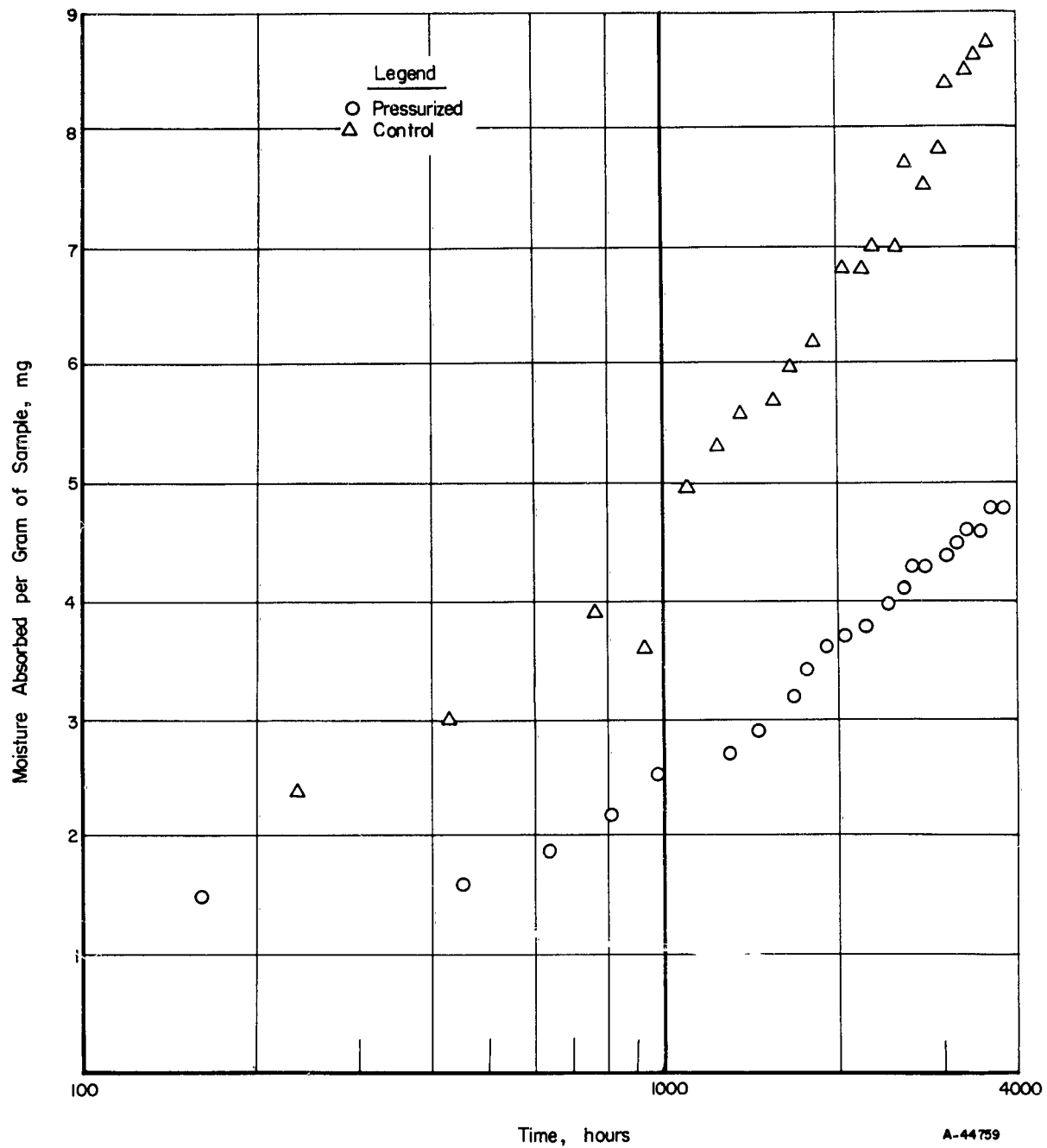


FIGURE 6. MOISTURE ABSORPTION OF 1-INCH, FILAMENT-WOUND OPEN CYLINDERS

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### Mechanical Testing

Several samples were removed from each of the various groups of cylinders undergoing immersion tests. These were tested in a standard Baldwin testing machine, using compressive loads to determine the axial compressive yield stress and also an empirical flexural strength. These data are summarized in Table 1. It will be noted that there is a definite hydrostatic-pressure effect associated with deterioration in mechanical properties. This is perhaps not as evident in the axially measured yield strength as it is in the radial load. The values of maximum radial load, which were determined in a "modified" flexural test, would appear to indicate that moisture is collecting at the resin-glass interface.

When conducting the radial compression of various exposed and unexposed cylinders, a difference was observed in the audible sounds occurring after yield. These sounds were recorded for both unexposed and exposed samples by means of a contact transducer and an Ampex Model 601 tape recorder. On playback, it was observed that high-intensity sounds occurred more often in the unexposed samples. At lower tape speeds, various high frequencies became audible. Again, these sounds were of relatively greater intensity in the unexposed samples. It is not known whether the absence of sound from the exposed samples is due to fiber slippage in the samples, or to a damping action due to the presence of moisture in voids at the resin-glass interface, or to a plasticizing action of the moisture on the resin.

Several\* of the neat resin cylinders being used in the immersion studies were sacrificed for the purpose of observing changes in compressive modulus. Such a change would be indicative of a plasticizing action on the neat resin by the water. However, such slight differences were observed that they could conceivably be due to normal variation between samples.

The diffusion of water in the neat resin, however, has been observed to be an extremely slow yet real phenomenon. Considering this, it is likely that the greater portion of the resin in these test cylinders contains little or no moisture, i. e., moisture has penetrated only a little way into the plain resin. The compressive-test figures shown below for capped, 3/4-inch, neat resin cylinders, then, are essentially those for unexposed resin.

	Unexposed		Exposed	
	9350	9350	8560	8940
Yield Stress, psi				
L/D Ratio	1	1	1	1
Time at 10,000 PSI, hr	0	0	2100	2100

This behavior is in sharp contrast to that observed with the same resin when reinforced with glass fiber. Also, these results emphasize the need for careful study of the composite and particularly of interfacial effects. A series of hardness measurements is consequently planned for both unexposed and exposed resin cylinders prepared simultaneously.

To determine if the moisture causes debonding, a series of flex bars is being exposed to water at high pressures and at ambient pressure. By observing any resulting change in flexural modulus, the effects of exposure should become apparent.

\* Only two samples remained which had no evidence of crazing in the high-pressure environment. These were removed for testing.

TABLE 1. MECHANICAL PROPERTIES OF EXPOSED AND UNEXPOSED FILAMENT-WOUND CYLINDERS

	Unexposed Cylinder	Open Control		Capped Cylinder Exposed at 10,000 Psi
		Cylinder Exposed at Atmospheric Pressure	Open Cylinder Exposed at 10,000 Psi	
1 % Offset Yield Stress in Axial Compression, psi	30,729	22,395	20,833	21,153
Maximum Radial Load, pounds	3,120	2,700	1,800	2,100
Duration of Exposure, hours	--	2,300	2,300	2,300
L/D Ratio	1	1	1	1
Winding Pattern	Hoop	Hoop	Hoop	Hoop
Number of Cylinders Tested for Each Value Shown	2	2	2	1

### TENTATIVE THEORY OF MOISTURE ABSORPTION

On the basis of the experimental evidence, the water-absorption phenomenon in glass-reinforced structures may be tentatively described.

Weight-gain experiments as well as interferograms of immersed epoxy resin films demonstrate that water may diffuse at a very low rate through a cured epoxy resin. The rate of diffusion appears to be independent of the hydrostatic pressure of the immersion medium. No evidence of a change in properties of the neat resin is yet attributable to the presence of water.

In glass-containing samples, however, the characteristic decrease in compressive yield stress may indicate (a) plasticization of the resin by absorbed water or (b) a weakening or destruction of the resin-glass bond. Experiments are now in progress to determine which of these factors may account for the reduction in properties. Mechanical tests on cylinders immersed at atmospheric and high pressures indicate that this reduction occurs more rapidly in the latter group.

Moisture may also "enter" the laminate by flaws due to improper cure to prolonged loading at high pressures. The latter have been observed by means of electron microscopy. Such flaws would allow more immediate access of the water to interlaminar flaws and potential film formation at the resin-glass interface. Experiments are now in progress to detect the collection of water at the resin-glass interface.

### FUTURE WORK

In the coming quarter, diffusion measurements will be completed on the hydrophilic model systems previously mentioned. In addition, a prolonged series of diffusion measurements will be attempted on cured epoxide resins.

In the second phase of this program, immersion experiments will be continued with those remaining samples. In addition, a study of similar samples immersed in sea water has been started.

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